Ground-Based Experiments in Support of Microgravity Research Results - Vapor Growth of Organic Nonlinear Optical Thin Film

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Abstract

A versatile flight qualified Moderate Temperature Facility (MTF) has been developed for materials processing in space. The MTF is capable of growing crystals and thin films on Earth and in space on a wide variety of carriers including the Space Station. The four successful flights on the U.S. Space Shuttles demonstrated the MTF materials processing capability. Two growth cells on the Consortium Complex Autonomous Payload (CONCAP) IV-02 on STS-59 and eight growth cells on CONCAP IV-03, STS-69, were devoted to the vapor growth in space of an organic nonlinear optical (NLO) material, N, N-dimethyl-p-(2, 2-dicyanovinyl) aniline (DCVA). Crystal growth experiments on the ground had always yielded small bulk crystals of DCVA. The space grown samples, however, showed a completely different growth form than did the ground samples. Instead of the bulk crystals all of the space samples were thin films with ordered and oriented structure deposited on the disordered substrates. The CONCAP IV-03 flight results extend, confirm, and are consistent with those of the CONCAP IV-02 flight. These results are particularly important because thin films lend themselves to a whole new range of applications in addition to those associated with bulk crystals. To date, the vapor-deposited DCVA thin films have never been obtained on Earth. A ground-based program is proposed to explore possible approaches to grow on Earth, from vapor, the DCVA thin films. Vapor pressure measurements, at various temperatures, of DCVA and other related NLO organic materials, for a reliable evaluation of the relative weight of the convective and diffusive flux contribution, will be performed. Vapor growth experiments altering the MTF's orientation to the gravity vector, changing the substrate temperature, modifying the temperature profile at many combinations of warm-up rates, measuring the background pressure in the MTF oven, will be carried out. The researchers hope to refine their results in a follow-up flight experiment, with electro-optical device-quality substrates so that the NLO effectiveness of these space-only grown films can be quantified.

The aim of the present work is to study and understand the macroscopic and microscopic terrestrial conditions under which the organic molecular DCVA and other related organic thin films can be reproducibly prepared and analyzed. The space-only grown DCVA thin films are an example of "van der Waals epitaxy" on inorganic substrates. Understanding this new growth process is vital if we are to fully exploit its potential to incorporate highly nonlinear optically active organic materials as active opto-electronic and electro-optic components in modern integrated logic device structures.

DCVA has been chosen for several reasons: it is, first of all, a fairly large organic molecule with sufficiently low room temperature vapor pressure, which is easily synthesized. Crystalline DCVA is an NLO material with good figure of merit and potential applications in the modern integrated logic devices. The material displays thermal stability with no glassy state, polymorphism or polymerization in the melting-solidification cycles. All these features are important to withstand many conventional semiconductor fabrication processes. The three year old space-only grown DCVA thin films proved to have thermodynamic and photochemical stability. Various parameters such as seeding, source temperature, transport flux, and substrate material were tested for their

effects on film growth. None appeared to strongly influence the growth of thin films. Ordered and oriented thin films have been grown on disordered copper substrates, as resulted from the DSC, FTIR and XRD spectra. Growth on aluminum substrates yielded isolated areas of non-uniform coverage. The best thin film samples have a smooth and featureless surfaces. A complete different microstructure morphology was observed on the edge regions.

In addition to carrying out a series of ground-based experiments aimed at understanding why the microgravity experiment results are so dramatically different from the results obtained when using the hardware on the ground, two changes need to be made to the experimental equipment. The first change is to install a fiberscope camera as part of the growth cell so that the growth of the crystals and thin films can be directly observed. At present all growth parameters are preset and nothing is known about the growth form until the experiment is complete. The ability to visualize the growth process in real-time is critical in coming to an understanding of this fundamental puzzle of why the growth form is so vastly different on Earth and in space. One barrier to using a fiberscope in the past has been that the fiberscope needs to operate in a 150 °C environment. Until recently the technology has not existed to do this. Fiberscopes are typically limited to long-term operating exposures of 90 °C. The technology now exists, however, to build the fiberscope we need. The second change is to upgrade the control electronics.